SYTHESIS OF ALIGNED CARBON NANOTUBES BY MICROWAVE PLASMA CHEMICAL VAPOR DEPOSITION

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Abstract

Aligned carbon nanotubes (CNTs) were synthesized on flat graphite substrates and silicon wafers by microwave plasma chemical vapor deposition (MPCVD) of a gas mixture of methane and hydrogen. Nickel catalyst was not directly deposited on the substrates, but migrated from a small piece of catalyst supplier to the substrate surface during microwave plasma pretreatment. SEM and TEM were used to characterize the morphologies of the CNTs.

INTRODUCTION

CNTs have many potential applications due to their superior mechanical, thermal, and electrical properties. Currently CNT synthesis methods include arc discharge, laser ablation, and chemical vapor deposition (CVD). The CVD method is used for rapid synthesis of nanotubes with high purity at lower temperatures and is easy to scale up for commercial production. Microwave plasma chemical vapor deposition (MPCVD) has been used to synthesize aligned nanotubes. These nanotubes are aligned either vertically to the substrate [1-2], or parallel to the substrate [3]. The alignment was affected by catalyst grain sizes, temperature, gas composition, etc. The CNTs produced in a batch MPCVD process was usually several tens of micrometers long with aspect ratio in the range of 300 to 1500. In this paper, we report the synthesis of high aspect ratio, up to 4500, and well-aligned CNTs with MPCVD method.

EXPERIMENTAL

The CNTs were prepared by MPCVD at a growth temperature range of 680 to 700^{o}C, with a pressure range of 4.7-4.9 kPa, and under the microwave power of 2.2 kW. The hydrogen flow rate was 80 sccm (Standard Cubic Centimeter per Minute) while the methane flow rate was 20 sccm. The growth time was 20 minutes. The silicon wafers were pretreated with diamond carbon. The nickel catalyst was not deposited onto the substrates or wafers, but migrated from a small piece of catalyst supplier onto the substrates during microwave plasma pretreatment. The catalyst supplier on the graphite substrate was silicon sputter deposited with a 30nm nickel layer. The detailed process is described in our patent application [4]. After synthesis, a piece of nanotube film was collected from the substrates and was examined with SEM and TEM

PRELIMINARY RESULTS AND DISCUSSIONS

Figure 1 shows 45° tilted SEM image of the synthesized CNTs on graphite substrate, which are 200-250 micrometer long and aligned vertically to the substrate surface, though slightly curly. The vertical alignment of nanotubes synthesized with plasma enhanced CVD was speculated to result from the electrical self-bias field generated on the substrate surface in plasma environment [1]. The plasma sheath may also influence the alignment of the nanotubes synthesized in these experiments. Figure 2 shows a typical TEM image of the CNTs. The diameters of the nanotubes are in the range of 40 to 70nm. They are not completely hollow and seem to be separated into many nano-compartments by curved platelets. The platelets were much thinner than the nanotube walls. Similar structures were observed in other studies [2, 5, 6]. The morphologies might be related with the nanotube growth mechanism. The nanotubes can grow from its root, and/or tip, depending on the positions of the catalyst particles. In this study, nickel was detected at the top of nanotubes with Energy Dispersive x-ray Spectrometer (EDS), indicating a tip growth route. The most widely accepted growth model of carbon nanotubes was adapted from the catalytic synthesis

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of carbon fibers. The hydrocarbon, first, decomposes into carbon and hydrogen at the front (exposed) face of the catalyst particle. Then, the carbon dissolves in the catalyst, diffuses through the particle, and precipitates at the trailing face to form the nanotube [7, 8]. The morphology results of the CNTs on silicon wafers are under characterization.

PRELIMINARY CONCLUSIONS

A MPCVD method can be used to synthesize CNTs on graphite substrates and silicon wafers. The nanotubes growth was catalyzed by nickel, which migrated onto the substrates and silicon wafers during microwave plasma pretreatment. For the CNTs on graphite substrates, SEM study showed that the CNTs were aligned vertically to the substrate surface and were up to 250 micrometers long; TEM results showed that the nanotubes had diameters in the range of 40 to 70 nanometers; The nanotubes were not completely hollow and were separated into many nano-compartments by curved platelets.

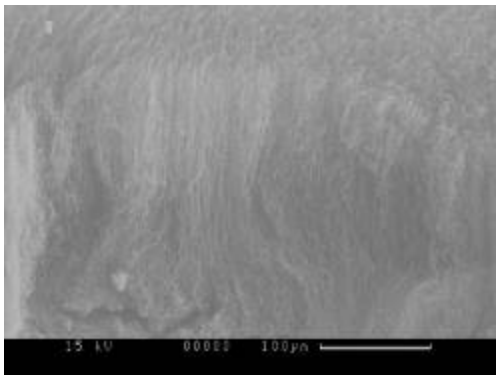

Figure 1. SEM image (tilted 45º) of the CNTs grown on a graphite substrate. The Scale bar is 100 micrometers.

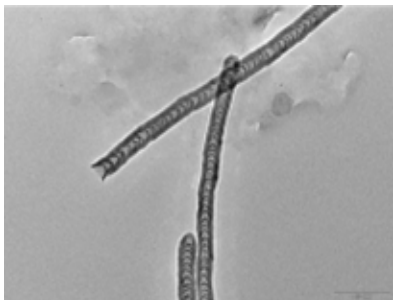

Figure 2. TEM image of the CNTs on a graphite substrate. The scale bar is 200nm.

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